IS 1251 (1988): Zinc Phosphide, Technical [FAD 1: Pesticides and Pesticides Residue Analysis]

BLANK PAGE

AMENDMENT NO. 1 MAY 1991
TO
IS 1251 : 1988 SPECIFICATION FOR ZINC PHOSPHIDE, TECHNICAL

(Third Revision)

[*Page* 1, *Table* 1, *Sl No.* (ii), *col* 3] — Substitute, '60·0' *for* '70·0'.

(*Page* 3, *clause* **A-3.1**, *first sentence*) — Substitute the following for the existing matter:

'Measure 100 ml of standard sodium hydroxide solution into the gas wash bottle (absorption bottle) *E*, 100 ml of standard potassium permanganate solution in gas wash bottle.'

(*Page* 3, *clause* **A-3.1**, *second sentence*) — Substitute the following for the existing matter:

'Measure 50 ml of standard potassium permanganate solution into each of the gas wash bottles *G* and *H*.'

(*Page* 3, *clause* **A-3.2**, *line* 8) — Substitute the following for the existing matter:

'Continue the reaction for at least 90 minutes.'

(FADC 1)

Printed at Progressive Printers, Delhi, India

UDC 632·958·31 : 661·847·451 (First Reprint FEBRUARY 1991) IS : 1251 - 1988

Indian Standard Reaffirmed 2009

SPECIFICATION FOR ZINC PHOSPHIDE, TECHNICAL

(*Third Revision*)

Pest Control Sectional Committee, AFCDC 6 [Ref : Doc : AFCDC 6 (2904)]

0. Foreword

0.1 In the preparation of this standard, due consideration has been given to the provisions of the *Insecticides Act,* 1968 and rules framed thereunder. However, this standard is subject to the restriction imposed under these regulations, wherever applicable.

0.2 Zinc phosphide, technical, is widely used as rodenticide for the control of bandicoots, rats and mice. The material acts as a stomach poison and on coming in contact with gastric juices in the stomach, produces phosphine gas which kills the rodents.

1. Scope — This standard prescribes the requirements and the methods of sampling and test for zinc phosphide, technical.

2. Requirements

2.1 *Description* — The material shall be dark-grey powder with a characteristic garlic odour. It shall be fine, heavy, free flowing powder free from lumps.

2.2 The material shall also comply with the requirements given in Table 1.

2.3 *Freedom from Sulphides* — The material shall be free of sulphides when tested by the method prescribed in Appendix C.

TABLE 1 REQUIREMENTS FOR ZINC PHOSPHIDE, TECHNICAL

(*Clause* 2.2)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Appendix of This Standard	Cl No. of IS : 6940-1982*
(1)	(2)	(3)	(4)	(5)
i)	Zinc phosphide (Zn_3P_2), percent by mass, *Min*	80·0	A	—
ii)	Zinc content, percent by mass, *Min*	70·0	B	—
iii)	Sieving requirements:	99·0	—	12·1
	a) Material passing through 150-micron IS Sieve [*see* IS : 460 (Part 1)-1985†], *Min* (*see also* Note 2), and			(*see* Note 1)
	b) Material passing through 106-micron IS Sieve [*see* IS : 460 (Part 1)-1985†], *Min* (*see also* Note 2)	92·0	—	12·1 (*see* Note 1)

Note 1 — For sieving requirement, start with 50 g of the material.

Note 2 — BS Sieves 100 and 150; ASTM Sieves 100 and 140; and Tyler Sieves 100 and 150 have their aperture within the limits specified for 150-micron IS Sieve and 106-micron IS Sieve respectively and may be used.

*Methods of test for pesticidies and their formulations (*first revision*).

†Specification for test sieves: Part 1 Wire cloth test sieves (*third revision*).

3. Packing and Marking

3.1 *Packing* — The material shall be packed according to the requirements given in IS . 8190 (Part 1)-1980 'Requirements for packing of pesticides: Part 1 Solid pesticides (*first revision*)', and IS : 8190 (Part 3)-1979 'Requirements for packing of pesticides: Part 3 Household pesticides'.

Adopted 21 March 1988 Gr 3

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

3.2 *Marking* — The containers shall be marked legibly and indelibly with the following information, in addition to any other information required under *Insecticides Act* and rules:

a) Name of the material;
b) Name of the manufacturer;
c) Date of manufacture;
d) Batch number;
e) Net mass of the contents; and
f) Minimum cautionary notice worded as in the *Insecticides Act* and rules.

3.2.1 *Standard marking* — Details available with the Bureau of Indian Standards.

4. Sampling — Representative samples of the material shall be drawn as specified in IS : 10946-1984 'Methods for sampling of technical grade pesticides'.

5. Tests — Tests shall be carried out in accordance with **2.1, 2.2, 2.3** and col 4 and 5 of Table 1.

5.1 *Quality of Reagents* — Unless specified otherwise, pure chemicals and distilled water [*see* IS : 1070-1977 Specification for water for general laboratory use (*second revision*)] shall be employed in tests.

Note — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

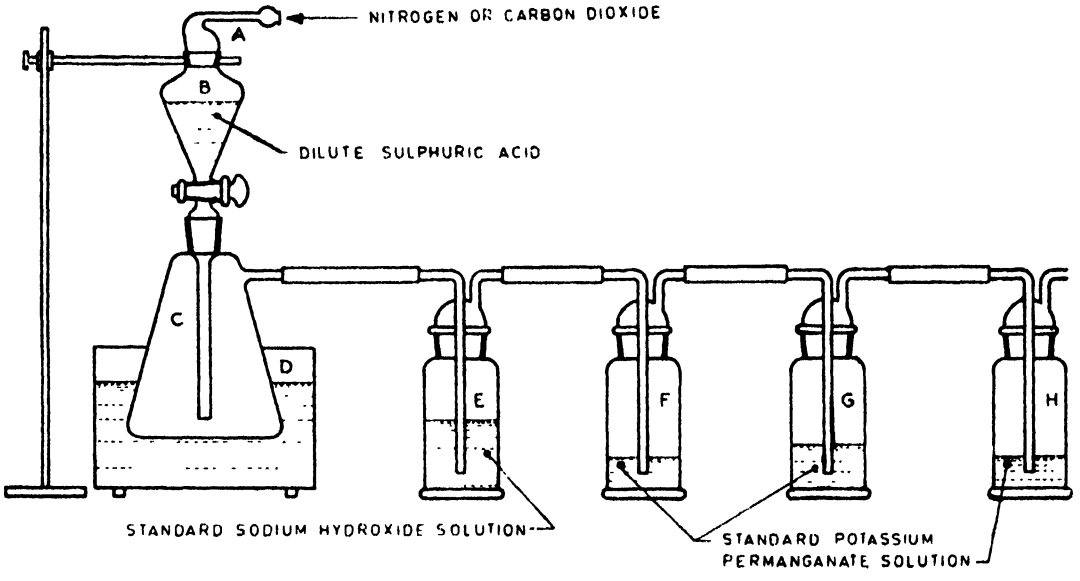
[*Table* 1, *Item* (i)]

DETERMINATION OF ZINC PHOSPHIDE (Zn_3P_2) CONTENT

A-1. Apparatus

A-1.1 The assembly of the apparatus is shown in Fig. 1.

A-1.2 The apparatus consists of a 250-ml reaction flask *C* with a standard interchangeable socket. A 250-ml separating funnel *B* and delivery tube (PVC tube surgical soft type) ase to be connected according to Fig. 1. The side tube is serially connected with four 200/250-ml absorption bottles *E, F, G* and *H* respectively. To the mouth of the separating funnel is attached a nitrogen or carbon dioxide inlet tube *A* as indicated in Fig. 1. The reaction flask is so mounted on a stand where it is possible to immerse it in the water bath *D* maintained at a temperature of 65 ± 5°C.



A = Adapter tubing (for nitrogen or carbon dioxide gas)
B = Separating funnel conical shape with cone and socket 250-ml
C = Reaction flask (Buchner) 250-ml
D = Thermostatically controlled water-bath
E, *F*, *G*, *H* = Gas wash (absorption) bottles (200/250-ml)

FIG. 1 ASSEMBLY OF APPARATUS FOR THE DETERMINATION OF ZINC PHOSPHIDE AND DETECTION OF SULPHIDES

A-2. Reagents

A-2.1 *Standard Potassium Permanganate Solution* — Approximately 0·5 N.

A-2.2 *Sulphuric Acid* — 10 percent (*m/v*).

A-2.3 *Nitrogen Gas or Carbon Dioxide Gas* — From a cylinder.

A-2.4 *Sulphuric Acid* — 1 : 1 (*v/v*).

A-2.5 *Standard Sodium Hydroxide Solution* — 1 N.

A-2.6 *Standard Oxalic Acid Solution* — Approximately 0·5 N and acidified with sulphuric acid. Weigh accurately about 15·8 g of oxalic acid ($H_2C_2O_4.2H_2O$) and dissolve in about 200 ml of water contained in a 500-ml volumetric flatk. Add to the volumetric flask 125 ml of sulphuric acid 1 : 1 (*v/v*), make up the volume with water and mix.

A-3. Procedure

A-3.1 Measure 100 ml of standard sodium hydroxide solution into the gas wash bottle (absorption bottle) *E*, 100 ml of standard potassium permanganate solution into gas wash bottles *F* and *G*. Measure 50 ml of standard potassium permanganate solution into gas wash bottle *H*. Assemble the apparatus as shown in Fig. 1 (without dilute sulphuric acid in the separating funnel *B*). Pass the gas (nitrogen or carbon dioxide) slowly through the apparatus so as to displace air. Weigh accurately about 0·5 g of the material and transfer it into the reaction flask *C* quantitatively.

A-3.2 Disconnect the nitrogen gas tube and place 100 ml of dilute sulphric acid in the separating funnel *B*. Connect the nitrogen gas tube to the funnel again. Add dilute sulphuric acid (*see* **A-2.2**) to the reaction flask slowly and very cautiously drop by drop in the beginning, carefully regulating the rate of addition in such a way that a steady stream of bubbles appears in the gas wash bottles. When the addition of dilute sulphuric acid is complete, adjust the pressure of nitrogen gas or carbon dicxide gas so that a steady flow of bubbles is maintained in the reaction flask and the gas wash bottles. During this process, immerse the reaction flask *C* in the water bath maintained at a temperature of 65 ± 5°C. Continue the reaction for at least one hour. Sweep the last traces of phosphine from the flask with more rapid stream of nitrogen or carbon dioxide for at least 5 minutes. At the end of the reaction and sweeping period, disconnect the apparatus and quantitatively transfer the redused potassium permanganate solution contained in the three gas wash bottles *F*, *G* and *H* to a 1 000 ml or a convenient size beaker. Rinse the gas wash bottles and connecting tubes with 200 ml of standard oxalic acid solution, taking care to dissolve all the manganese dioxide. Add the rinsings to the reduced potassium permanganate solution contained in the beaker. Rinse the gas wash bottles and connecting tubes with water and transfer the rinsings to the same beaker. Warm the contents of the beaker to approximately 60°C and titrate the excess oxalic acid with standard pctassium permanganate solution.

A-3.3 Retain flask *C* for zinc determination as prescribed in Appendix B and gas wash bottle *E* for detection of sulphides as prescribed in Appendix C.

A-4. Calculation — Calculate zinc phosphide content of the material as follows:

Zinc phosphide (Zn_3P_2)

$$\text{content, percent by mass} = \frac{1{\cdot}613\,[\,(200 + A)\,N_1 - 200\,N_2\,]}{M}$$

where

A = volume, in ml, of standard potassium permanganate solution required for the titration of excess oxalic acid;

N_1 = normality of standard potassium permanganate solution;

N_2 = normality of standard oxalic acid solution; and

M = mass, in g, of the material taken for the test.

APPENDIX B

[*Table* 1, *Item* (ii)]

DETERMINATION OF ZINC CONTENT

B-1. Reagent

B-1.1 *Sodium Hydroxide* — 1 N.

B-1.2 *Disodium Ethylenediamine Tetra-Acetate (EDTA) Solution* — 0 1 N. Dissolve 18 6 g of disodium ethylenediamine tetra-acetate dihydrate in water and make up the volume to 1 litre in a volumetric flask. Standardize according to the procedure given in **B-2**

B-1.3 *Standard Zinc Solution* — Dissolve 3 269 g of analytical reagent grade zinc metal in 20 ml of hydrochloric acid (1 : 1 *v/v*) and make up to 1 000 ml in a volumetric flask

B-1.4 *Erichrome Black T-Indicator* — Mix thoroughly 1 g of erichrome black T with 100 g of sodium chloride

B-1.5 *Buffer Solution* — Dissolve 67 5 g of ammounium chloride in 300 ml water, add strong ammonia solution (570 ml) and make it to 1 000 ml with distilled water

B-1.6 *Sodium Hydroxide* – 0 5 N

B-1.7 *Sulphuric Acid* — 10 percent (*v/v*).

B-1.8 *Ammonia Solution* — 10 percent (*v/v*)

B-2. Standardization of EDTA Solution — Pipette out 25 ml of standard zinc solution (**B-1.3**) in a 250 ml Erlenmeyer flask Neutralize it with 1 N sodium hydroxide Add 5 ml of a buffer solution (*see* **B-1.5**) and few specks of erichrome black T Titrate with EDTA solution. The end point is indicated by a sharp colour change from red to blue

One ml of 0 1 N EDTA solution 3 269 mg of pure zinc

B-3. Procedure — Filter retained solution (*see* **A-3.3**) and make up to 250 ml in standard volumetric flask. Take 25 ml aliquot in 500 ml Erlenmeyer flask Add 1 N sodium hydroxide till brown precipitate of iron is complete Then add 25 ml of sodium hydroxide Filter the solution and wash the precipitate with dilute sodium hydroxide solution (10 ml) twice Wash with distilled water (10 ml) Make filtrate solution neutral to litmus paper with dilute sulphuric acid, make it alkaline with dilute ammonia (*see* **B-1.8**) and add 10 ml buffer solution Titrate with EDTA solution using erichrome black T-indicator (few specks) till clear blue end point

B-4. Calculation — Calculate zinc content of the material as follows.

$$\text{Zinc content, percent by mass} = \frac{32\,69 \times V \times N}{M}$$

where

V = volume, in ml, of EDTA solution required for 25 ml aliquot in **B-3**;

N = normality of EDTA, and

M = mass, in g, of the material taken for test (*see* **A-3.1**).

APPENDIX C

(*Clause* 2.3)

DETECTION OF SULPHIDES

C-1. Reagent

C-1.1 *Standard Neutral Cadmium Sulphate Solution* — 2 M.

C-2. Procedure — At the end of the reaction and sweeping, disconnect gas wash bottle *E* (*see* **A-3.2** and **A-3.3**). Add 10 ml of standard neutral cadmium sulphate solution (*see* **C-1.1**) into the gas wash bottle *E* No yellow precipitate shall be formed. Formation of yellow precipitate indicates presence of sulphide (cadmium sulphide).

EXPLANATORY NOTE

This standard was first published in 1958 and subsequently revised in 1973 and 1984. It has been revised again to update its requirements.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed and calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Printed at Central Electric Press Delhi-28